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Caustic Embrittlement of Boiler Plate

A COMPREHENSIVE review of the present state of knowledge of the caustic embrittlement of boiler plate steel has recently been published by G. T. Athavale[1] in a paper which contains ninety-eight references to work on the subject. It is of interest to compare it with a paper, read a few weeks previously, by Dr. C. H. Desch[2] to the North-East Coast Institution of Engineers and Shipbuilders on intercrystalline cracking of boiler plates. Both authors recognise that caustic embrittlement and intercrystalline cracking are not the same thing; but, in spite of that, much of the same ground is covered in the two papers, though in some of the final conclusions there is considerable divergence of view. Whereas Desch insists that caustic embrittlement is an incorrect term because the steel between the cracks is not necessarily brittle, Athavale, though claiming that the steel may become brittle without any visible change in appearance, seems to assume that embrittlement necessarily precedes intercrystalline cracking. He refers to, and illustrates, three types of cracks which may occur in boilers :—

(1) Corrosion fatigue cracks which are present as a result of electrochemical processes rendered important by the presence of Cl', NO'_3, or SO''_4. They follow the lines of stress without regard to grain boundaries.

(2) Ordinary fatigue cracks which are caused purely by mechanical stress and follow an intracrystalline path.

(3) "Brittleness cracks" which are recognised by their intercrystalline path. These are the cracks occurring, in the author's view, as a result of caustic embrittlement.

Having linked up intercrystalline cracking with embrittlement in this way, Athavale proceeds to discuss the causes of caustic embrittlement and examines the suggestions that alkali is not the chief cause of the embrittlement, but rather the type and quality of the steel and the local stresses which are present in the boiler; that caustic embrittlement depends on hydrogen absorption; and that the embrittlement depends on precipitation-hardening phenomena due to the presence of nitrogen in the steel. He comes to no certain conclusions about the cause of the phenomenon, but is able to state definitely that for the occurrence of caustic embrittlement the simultaneous presence of alkaline liquid and mechanical stress (probably up to the yield point of the steel) is necessary. The immediate cause of the embrittlement is held to be hydrogen absorption. This is supposed not only to diminish the strength of the steel, but to react with certain impurities present in the steel in such a way as to promote disruption between the crystal grains. A concentration of alkali suitable for reaction with iron is not reached in the boiler, but the hydrogen absorbed is formed by reaction of the steel with the highly concentrated alkali solution which may be found in the riveted joints or boiler seams.

In his claim that stress and the presence of alkali are essential, Athavale is on sure ground; in the conclusion that hydrogen plays an important part in the production of cracks he is also probably correct, though, as he admits, the mechanism of the action is not known with certainty; but he then goes on to introduce suggestions that are of a much more highly speculative character, with reference to the properties of the steel.

There has been a tendency in Germany to hold the steel responsible for the failures of boiler plates and to insist that, to be satisfactory, the steel must be specially resistant to corrosion attack under the extreme conditions of temperature and pressure that it is called upon to withstand. The alloy steels being ruled out on the grounds of expense, attention was directed to certain killed or aluminium-containing steels which were held to be more corrosion resistant than semi-killed or rimming steels. Considerations of this kind led to the development of Izett non-ageing steel, which was claimed to be immune. The claim that Izett and aluminium steel show a lesser tendency to become brittle and that steels of high nitrogen content, such as Bessemer steels, show a greater tendency is repeated by Athavale; and, governed by the idea that boiler cracking is a sequel to embrittlement, he adds to his conclusions two statements which concern the steel. These are that the type and quality of the boiler steel has a great influence in the progress of caustic embrittlement, those steels containing intercrystalline cavities (that is, unkilled steel) being most susceptible; and that high-nitrogen steels incline so strongly to brittleness after cold working and ageing that the presence of nitrogen, capable of being precipitated, can also be regarded as an important cause of caustic embrittlement. These conclusions may be questioned. It is by no means shown by the evidence brought forward that intercrystalline cracking is dependent on susceptibility to ageing embrittlement. On the other hand, it cannot be denied that the primary cause of ageing, viz., appreciable permanent deformation of the metal, is present round most holes. Riveted boilers of good, as well as of poor, construction may show a tendency towards brittleness in the vicinity of the rivet holes as a result of ageing, with possible intensification of the embrittling effect produced by the chemical action of the boiler water on the steel. On the subject of the embrittlement of boiler steel, the correlated abstract of E. P. Partridge and W. C. Schroeder[3] still

remains the standard of reference down to 1935, and it may be well, with these authors, still to regard ageing as a contributory factor in causing embrittlement failures, though its importance is probably not as great as at one time was supposed.

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[2] Reported in THE ENGINEER, March 31st, 1939, page 418.
[3] *Metals and Alloys*, 1935 (June to December).

The Influence of Rate of Cooling on the Transformations in Steel

IN order to utilise the equilibrium diagram of the iron-carbon alloys as a guide to the heat treatment of steels and to the interpretation of microstructures, it is necessary not only to study equilibrium conditions, but also to know what displacements or alterations the transformations indicated by the diagram undergo as a result of increasing the rates of cooling the steel. The hardening process is dependent on undercooled transformations, the downward displacement of which may be investigated in two ways. The first and older method is to study the quenching process by taking cooling curves at different rates of cooling. This method was first used by H. Le Chatelier. It was developed by A. Portevin and his collaborators, and also by F. Wever and N. Engel.[1] The cooling curves taken at different rates show the displacement of the temperature of transformation as the rate of cooling increases. The second method consists in rapidly cooling the austenite solid solution to various sub-critical temperatures, and then following the course of the transformation by recording the alteration of certain physical properties. This method, used by E. S. Davenport and E. C. Bain, has also been developed by F. Wever and H. Lange.[2] By correlating the velocity of transformation, structure, hardness, and other properties, this method has proved to be particularly useful in recording the changes which the transformation process undergoes at gradually lower transformation temperatures.

A large amount of work has been done at the Kaiser-Wilhelm Institute, Düsseldorf, involving both quenching experiments and experiments at constant temperature. That recently published under the general title "The Influence of Cooling Velocity on the Transformations of Steels," refers to data derived from heating and cooling curves. The material employed in this work consisted, in addition to a specimen of electrolytic iron containing 0·02 per cent. of carbon, of a series of carbon steels containing 0·06 to 1·75 per cent. of carbon, with phosphorus 0·01, sulphur 0·010 to 0·018, and manganese 0·11 to 0·14 per cent. The silicon content was under 0·06 per cent. in the medium and low carbon steels and 0·10 to 0·12 per cent. when the carbon was 0·75 per cent. or more.

The first contribution, by F. Wever and A. Rose,[3] describes a new method for the study of the transformation processes at high rates of cooling, and the influence of rate of cooling on the transformations of carbon steels. This was based on that of Wever and Engel, who used as a specimen an electrically heated wire, cooled by a stream of hydrogen, the rate of cooling being determined by the velocity of the stream and diameter of the wire. This had the disadvantage that the specimen available for hardness testing and microscopical examination was very small, and that the range of compositions which could be drawn into wire was very limited. While hydrogen cooling was retained by Wever and Rose, the specimen used was changed to a disc, 0·5 mm. thick and 5 mm. diameter, which was welded to a thermo-element of 0·3 mm. diameter wire. Cooling velocity was standardised in relation to manometer readings at different positions in the hydrogen stream. By the aid of an electro-cardiograph a temperature record was obtained on a fixed sensitised plate. The blackening of the plate, which was determined photometrically, corresponds directly to the reciprocal of the angular velocity of the mirror of the cardiograph and therefore to the reciprocal of the rate of change of temperature. Curves of temperature plotted against rate of change of temperature revealed the temperatures at which transformation took place. The rate of cooling was expressed as the coefficient k in the expression of Newton's law of cooling

$$d\theta/dt = -k(\theta - \theta_0).$$

Thus a rate, for example, of $k = -0{\cdot}5$ would be 500 deg. per second at about 1000 deg. Cent., or 250 deg. per second at about 500 deg. Cent. The transformations in carbon steels fall discontinuously, in three stages, to lower temperatures as the rate of cooling is increased. The behaviour of the steels can be simply and clearly represented by a diagram (Fig. 1), in which three undercooled transformation

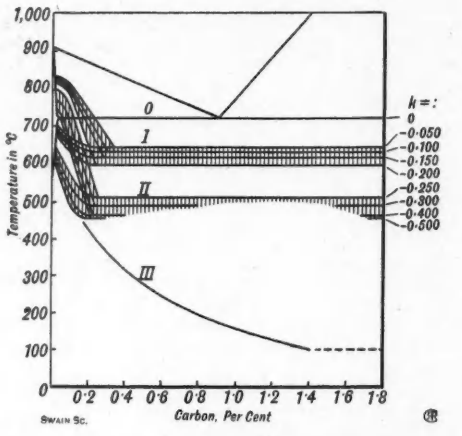

FIG. 1—*The Undercooling Diagram of Carbon Steels*
Rate of cooling at temperature $\theta = -K \cdot \theta$ *(approximately)*

temperatures, or regions of temperature, are shown. The equilibrium diagram itself gives the transformation temperature under ideal conditions of slow cooling. The undercooling diagram 1 shows the pearlite change occurring at about 650–600 deg. Cent. at rates $k = -0{\cdot}05$ to $-0{\cdot}20$. The carbon content of the apparent eutectoid is lowered to 0·4 per cent. and pre-eutectoid cementite separation is suppressed. The structure produced when transformation occurs in this first undercooling stage is ferrite and sorbitic pearlite up to 0·4 per cent. of carbon, and with higher carbon content a sorbitic structure only. At higher rates of cooling $k = -0{\cdot}25$ to $-0{\cdot}50$, the first undercooling diagram is replaced by the second, in which the transformation occurs at 500 deg. Cent. or below. The apparent eutectoid now contains

0·35 per cent. of carbon. The austenite transformation no longer occurs completely at this temperature in all compositions, and hence depression to the second stage involves also the appearance of the third (or martensitic) stage, while the structure at the same time changes from ferrite and sorbitic pearlite, through troostite and martensite to pure martensite. In steels with less than 0·35 per cent. of carbon, the transition occurs over a broad range of cooling velocities ; in steels of higher carbon content, on the other hand, and especially in hyper-eutectoid steels, the transition to martensite occurs abruptly at a rate of cooling of about $k = -0·3$ or less. In steels with 1·2 to 1·5 per cent. of carbon, this critical rate falls to $k = -0·23$ or $-0·24$. The transition from the equilibrium condition to the undercooled stages 1, 2, and 3, is, of course, characterised by changes in

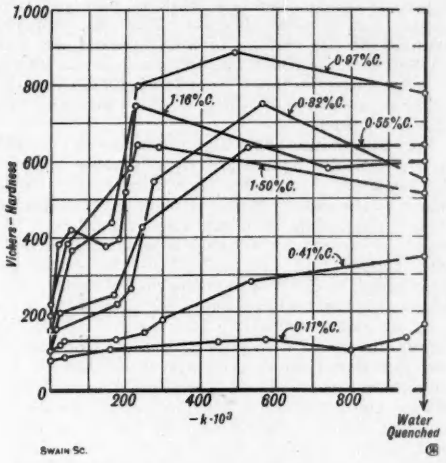

FIG. 2—*Vickers' Hardness of Carbon Steels in relation to Rate of Cooling*

mechanical properties which are indicated in the hardness diagram (Fig. 2).

It is clear from a diagram such as Fig. 3, which indicates the displacement of the transformation temperature of a steel containing 0·41 per cent. of carbon, that a rate of cooling of $k = -0·025$ depresses Ar_3 to the temperature of Ar_1, and a rate of $k = -0·050$ lowers the pearlite point about 80 deg. with consequent effect on the structure and properties ; while the influence of similar rates of cooling on the properties of the high-carbon steels is, as will be seen from Fig. 2, even more pronounced.

In the second section of the report F. Wever and A. Rose[4] describe attempts to determine more accurately this relatively great influence of slow rates of cooling on the process of transformation. In order to follow the changes which occurred at the slowest possible rates use was made of an electrically recording dilatometer of great sensitivity so that small specimens, the temperature of which was continuously recorded, could be employed. The dilatometer was arranged to deal with rates of cooling of 1, 15, and 40 deg. per second. In a third section of the work H. Lange[5] described a vacuum dilatometer in which the length changes were measured by the deflection of a beam of light and were recorded by means of a photo-electric device. This enabled much slower rates of cooling to be used and Lange's main results covered the range 0·5 to 8 deg. per minute.

The dilatation-temperature curves were thus determined for cooling rates from 0·01 to 40 deg. per

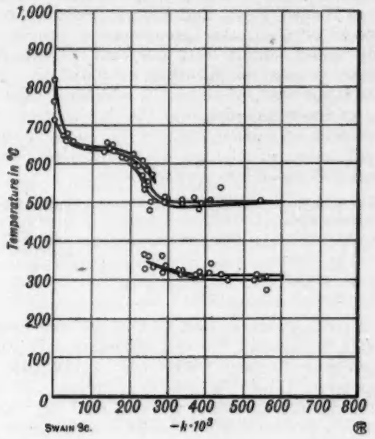

FIG. 3—*Transformation Temperatures in relation to Rate of Cooling of 0·41 per cent. Carbon Steel*

second. The results are summarised in undercooling curves, Fig. 4, which supplement the information given in the first section of the report and complete the data for the slower rates of cooling. These curves show that at the rates employed the undercooling of the pearlite change is practically independent of the carbon content. The Ar_3 transformation is consider-

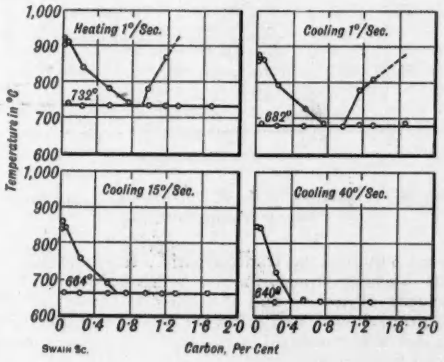

FIG. 4—*Transformation Diagram of Carbon Steels for Different Rates of Heating and Cooling*

ably more strongly undercooled than the Ar_1 change, and pre-eutectoid separation is suppressed before a rate of cooling of 15 deg. per second is reached. The dependence of the temperature of the pearlite transformation on the heating or cooling rates, expressed as degrees per second at 1000 deg. Cent. (that is,

as $-k.10^3$), is shown in Fig. 5. The data of Wever, Rose, and Lange are compared in the diagram with the results obtained by R. F. Mehl and C. Wells[6] on the superheating and undercooling of high-purity iron-carbon alloys made from hydrogen-purified carbonyl iron carburised in dipentene-hydrogen and dipentene-benzene-hydrogen mixtures. The Ac_1 and Ar_1 points of this series converged more nearly than those of Wever, Rose, and Lange, probably because the steels of the latter investigators contained, as already noted, about 0·12 per cent. of manganese, and some of them (with carbon over 0·7 per cent.) at least 0·1 per cent. of silicon in addition. The mean values of the temperature of the A_1 change (which is independent of carbon content) given by Lange for different rates of change of temperature between 600 and 950 deg. Cent. are as follows :—

Rate of change of temperature, deg. per min.	Temperature of Ac_1, deg. Cent.	Temperature of Ar_1, deg. Cent.
8	733	688
5·3	731	689
2·7	729	691
0·5	726	699

With a cooling rate as slow as 0·5 deg. per minute or 0·01 deg. per second, the transformation temperature is depressed 20 deg. below the equilibrium value extrapolated from the heating curves. It is not possible by extrapolating the Ac_1 and Ar_1 curves of Fig. 5 to arrive at any consistent conclusion as to the

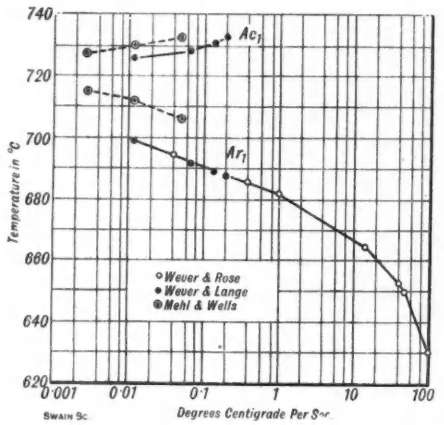

FIG. 5—*Temperature of the A_1 Transformation in relation to Rates of Heating or Cooling.*

exact equilibrium transformation temperature, *i.e.*, the limiting transformation temperatures for zero velocity of heating and of cooling do not agree. The diagram shows clearly that the rate of transformation in the neighbourhood of the equilibrium temperature must be extremely small.

This fact has an important bearing on the interpretation of heating and cooling curves, since these cannot be successfully obtained unless an appreciable difference of temperature exists between the specimen and its surroundings, involving rates of heating or cooling which may amount to 5 deg. per minute, and so accounting in fairly pure carbon steels (according to Lange's dilatometric curves) for a superheating of 5 deg. or an undercooling of 10 deg. compared with the values which would have been obtained if the temperature had been changing at one-tenth of the rate, *i.e.*, at 0·5 deg. per minute.

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2 *Ibid.*, 1932, 14, 71.
3 *Ibid.*, 1937, 19, 289.
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The Improvement of Metal Surfaces by Diffusion

ENGINEERING demands for uniform structural materials are met by choice of composition, method of production, hot and cold working, and heat treatment ; but it often happens that the properties required in the surface of a component are independent of those which must characterise its interior. Examples may be taken from the light alloys on the working properties of which so much attention is at present focused.

Pure aluminium has a high resistance to corrosion. It is practically unattacked by sea water, owing to the formation of an oxide layer which hinders further attack by the water. However, the great disadvantage of pure aluminium is its low strength, which is very largely overcome by alloying it with magnesium or with copper and magnesium, in the latter case (duralumin) at the cost of corrosion resistance. Thus arises a demand to combine resistance to corrosion with adequate strength. The method of cladding aluminium alloys with pure aluminium, for example, was introduced to resist surface corrosion. Pure aluminium sheet is placed over aluminium alloy sheet, and the composite sheet rolled in such a way that the final product has a thin adherent surface layer of pure aluminium. Another form of protection against corrosion is the oxide layer produced by electrolytic means on aluminium base alloys. These anodised surfaces possess other valuable properties, such as the possibility of dying them with various colours. The oxide layer is about 5 to 30 μ thick, and as it is penetrated by a system of very fine capillaries, a further treatment is necessary to fill up the pores. The modified Bauer-Vogel process (M B V process) is also used for surface protection, and depends likewise on chemical oxidation. In this the protective layer consists of the oxides of aluminium, chromium, &c., and is 1 to 2 μ thick. Protective paints are also used.

The surface hardness of a constructional material is of importance also, and it may be increased by "diffusion." The first example of this was the well-known case-hardening of iron, brought about by causing carbon to diffuse into its surface layers. It is important that both metals which take part in a diffusion process should form solid solutions with one another without the presence of an intermetallic compound. Three possibilities exist :—(1) Both metals form a continuous series of solid solutions with one another ; only solid solution occurs, therefore, on diffusion. (2) Both metals, besides forming mutual solid solutions α and β, also possess a range in which the two phases α and β exist together in the structure ; these, on diffusion, show a sudden change in concentration at the edge of the region of solid solution. (3) Both metals, besides forming solid solutions, form

intermetallic compounds, so that as many layers may exist between them as there are phases at the temperature of the experiment.

In general, the diffusion of a metal A into a metal B can be brought about by heating the metal B when embedded in a powder consisting of metal A. Further, Seith and Beerwald[1] found that heating a piece of aluminium in which a small piece of magnesium had been inserted, resulted in a microsection revealing four separate zones in the diffusion layer, the α, β, γ, and δ phase of the constitutional diagram. The phase between 45 and 46 per cent. of magnesium, newly discovered by Kawakami, was not observed. The presence of a particular phase depends on the speed of diffusion of the metal into the adjacent phase layer. Intermetallic compounds are not desirable for improving metal surfaces. It is only practicable to introduce magnesium up to the saturation point of the α-solid solution, since a higher magnesium content makes the surface very hard and brittle. The formation of brittle compounds unfortunately leads to flaking off of the surface on subsequent fabrication. To avoid this, inert substances, such as salts, are added to the powdered metal, so as to reduce the concentration of the diffusing metal at the surface to be treated, and better contact and cleaner surfaces are obtained.

Diffusion of one metal into another has been carried out by immersing a piece of the metal in a fused salt bath in which the diffusing metal is suspended in the form of a fine powder. The bath is kept in continual movement, so as to bring the powdered metal into contact with the surface of the piece. Iron or manganese alloys are said to be made rust-free and resistant to acid attack by impregnating their surface with silicon. It is claimed that iron, nickel, and copper may be surface-hardened by heating them in tungsten powder at 800 deg. to 1350 deg. Cent., at which temperature diffusion occurs. Iron or nickel surfaces are said to be improved by heating them in contact with powdered metal, such as copper, to which calcium nitrate, potassium nitrate, lead acetate, or calcium nitrite have been added. Iron surfaces are said to be improved by heating in the presence of gaseous chromium chloride, and this method has been tried to improve light metals. Aluminium surfaces have been heated at 250 deg. to 460 deg. Cent. in powdered magnesium to raise their hardness. At this temperature, below that of the eutectic, a surface alloy of aluminium and magnesium was formed, whose depth depended on the temperature and time of heating employed. In this case also a "thinner" consisting of magnesium oxide was added to the diffusion mixture, to give a more uniform surface hardening. Aluminium has been employed to improve iron surfaces, but owing to the relatively low melting-point of aluminium no diffusion occurs, so it has been proposed instead to use a higher melting aluminium-iron alloy as the cementing powder. Most of these methods suffer from the disadvantage that the presence of practically undesirable brittle intermetallic compounds cannot be avoided.

A method, based on the phase theory, is described by Seith and Jag,[2] whereby such brittle constituents are avoided. This is found to happen if the basis metal B is embedded in a powder consisting of a mixture of B with the diffusing metal A of such a composition that phase equilibrium could be reached between a solid solution of the basis metal B and the "alloy" powder. Aluminium, magnesium, and copper were chosen, and attempts were made to render them harder and more corrosion-resistant by exposing them to the diffusion of other metals in such a way that only a thin layer, consisting of α-solid solution, which possessed the necessary properties or could attain them as a result of precipitation hardening on cooling, was formed with the diffusing metal. For example, with aluminium and magnesium, when only powdered magnesium was used, undesirable intermetallic compounds, such as Al_2Mg_3 and Al_3Mg_2, could not be avoided in the surface layer produced. These layers were so brittle that they flaked off under the slightest force. Accordingly, the powdered intermetallic compound was used instead of the pure magnesium powder, *i.e.*, the phase in equilibrium at the diffusion temperature with the aluminium-rich α-solid solution of aluminium and magnesium. An alloy with about 38 per cent. of magnesium, corresponding to the β phase, Al_2Mg_3, was chosen for the magnesium-containing powder. The consequence was that the surface of the aluminium could take up, at the most, only as much magnesium as would be present in the saturated α-solid solution at the temperature of the experiment. Further, a smooth surface was formed on the specimen. In this way, aluminium, tin, bismuth, and copper were made to diffuse into magnesium as well as silicon, tin, beryllium, antimony, and phosphorus into copper.

In order to produce an α-solid solution by diffusion of magnesium into aluminium, rectangular blocks of pure aluminium were embedded in a magnesium-containing powder of composition equivalent to Al_3Mg_2 (the β phase), contained in a glass tube, from which the air could be extracted by a pump. A second tube contained a piece of aluminium embedded in pure magnesium powder. Both were placed together in a furnace and heated to near the eutectic temperature 451 deg. The test pieces had quite a different appearance after removal from the furnace. That embedded in the pure magnesium had a rough surface composed of aluminium-magnesium compounds, and had also increased in volume with its edges rounded. On the other hand, the one embedded in the Al_3Mg_2 had retained its size and shape as well as its original surface appearance. The surface layer was extremely brittle in the first case, and could be broken off in bits with a hammer, whereas in the second case the layer was not affected by such treatment. The depth of penetration of the magnesium into the aluminium was about 0·1 mm. A considerable increase in hardness of the superficial layers on the specimen occurred in all cases. Pure aluminium showed a hardness of 12·1 kilos. per square millimetre. After exposure to the diffusion at 445 deg. Cent. for sixty-two hours, the hardness rose to 87·2 kilos. per square millimetre by the Vickers test (hardened four-sided steel pyramid with a load of 0·3 kilos. for 30 seconds).

The diffusion of copper into aluminium and *vice versa* was investigated by Grube and Häfner,[3] using various methods. Copper was deposited electrolytically on the aluminium and the specimen heated to allow diffusion to occur. A mixture of copper chloride and copper powder was employed also, according to the equation

$$3\,CuCl_2 + 2\,Al = 3\,Cu + 2\,AlCl_3.$$

With this mixture cementation of the aluminium was carried out at 550 deg., 580 deg., and 600 deg., and an increase was found in the hardness of the aluminium. Similar experiments carried out with Al_2Cu powder showed that a clean surface resulted, whereas with copper powder the surface was blistered and grey. It was confirmed that below the eutectic

temperature at 500 deg. and 530 deg. no diffusion of the copper into the aluminium could be found. The variation with depth of copper concentration in the surface layer was estimated spectroscopically. The total depth of penetration was very small, 0·06 mm., at the most.

The diffusion of manganese into aluminium was investigated by means of aluminium specimens embedded in powdered Al_6Mn and heated *in vacuo* at 628 deg. for 87 hours. The corrosion resistance of pure aluminium was the best, but the experiments showed that the aluminium specimens containing diffused magnesium and manganese were considerably more resistant than duralumin, or than aluminium with diffused copper, the corrosion of which stood next to that of duralumin.

The diffusion of aluminium into magnesium was carried out by using powdered Al_2Mg_3 or pure aluminium powder. A clean surface was left afterwards on the test piece only when Al_2Mg_3 was employed. No increase in hardness was observed.

Diffusion of bismuth into magnesium was carried out by using powdered Bi_2Mg_3 as embedding material for the magnesium test piece, and a small increase in hardness of the magnesium was observed. Similarly, when Mg_2Cu was used as embedding material for magnesium, a similar increase in hardness occurred after heating for 48 hours at 468 deg. Cent.

The diffusion of silicon into copper was investigated by using powdered Cu_3Si as embedding material and heating at 760 deg. to 770 deg. The eutectic temperature of copper and Cu_3Si is 802 deg. No marked increase in surface hardness of the copper, as a result of silicon diffusion, was observed. In powdered silicon the copper test piece lost its shape completely when heated under similar conditions.

The diffusion of beryllium into copper was brought about by using powdered β-solid solution with 10 per cent. beryllium in copper as embedding material and heating *in vacuo* between 785 deg. and 832 deg. for periods varying from 43 to 118 hours. The surface hardness (Vickers) of the copper was considerably increased from 43·9 kilos. per square millimetre for pure copper to 81·6 kilos. per square millimetre after 118 hours' heating when embedded in the beryllium-containing powder. The beryllium content in relation to the depth of penetration was estimated spectroscopically. It fell from 9 per cent. at 0·05 mm. below the surface to 1·8 per cent. at 0·20 mm. and 0·5 per cent. at a depth of 0·5 mm. Satisfactory diffusion and a considerably greater increase in surface hardness was obtained for copper when heated in the presence of vaporised tin in a sealed tube (owing to the occurrence of sintering the embedding method proved unsatisfactory). The 23 per cent. copper-tin alloy was heated as vapour emitter, on the assumption that when equilibrium is attained at a constant temperature the vapour pressure of a metal in the α constituent is equal to its vapour pressure in the β-constituent. The copper specimen and the 23 per cent. copper-tin alloy were heated side by side *in vacuo*, and after absorption of the vapour until equilibrium was established there was a marked increase in surface hardness of the copper specimen which had maintained its size and shape perfectly after the treatment. The α phase was formed under equilibrium conditions with the vapour. Taking the Vickers hardness for pure copper as 43·9 kilos. per square millimetre, the surface hardness rose to a maximum of 91·5 kilos. per square millimetre after 62 hours' heating under exposure to the vapour at 748 deg. Cent. If pure tin was heated so as to vaporise instead of the copper-tin alloy, the pure copper specimen became grey in colour, and the concentration of the tin became high in the diffusion layer with formation of undesirable phases. Attempts to adjust the vapour pressure have been tried by heating the metal A to the diffusing temperature and the metal B to a temperature at which its vapour pressure corresponds to that in the desired surface layer. The diffusion of antimony into copper was carried out in a similar way by heating a copper-antimony alloy and exposing pure copper to its vapour. If pure antimony alone were heated, the copper specimen was destroyed.

Corrosion experiments carried out on the pure copper surface and on the alloy surfaces produced on the copper showed that the pure copper was the most resistant to corrosion by 3 per cent. hydrochloric acid, the next best resisting surface being that containing beryllium.

The utility of these alloy layers can only be assessed with reference to some specific requirement, but the principle of using an intermediate phase as the cementation medium is of general utility as a safeguard against the production of mechanically defective surfaces.

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Rhenium-Iron Alloys

ALTHOUGH rhenium was only discovered a few years ago, its chemical and physical properties have already been extensively studied. On the other hand, little is known of its alloying properties in relation to other metals. At first the extreme scarcity and high price of the metal made the study of alloys impracticable, but more recently it has become available in larger amounts, and although the study of rhenium alloys can, at the present time, have no technological importance whatever, it has a theoretical importance on account of the position of rhenium in the periodic table where it borders on some important alloy steel-forming elements as indicated below :—

Group and Atomic Number.

IV.	V.	VI.	VII.
22 Ti	23 V	24 Cr	25 Mn
40 Zr	41 Nb	42 Mo	43 Ma
72 Hf	73 Ta	74 W	75 Re

Hans Eggers has recently studied the niobium-iron diagram and compared the influence of niobium with that of its neighbours, zirconium, vanadium, tantalum, and molybdenum.[1] He has also extended his work to the binary system iron-rhenium.[2] The alloys were made from carbonyl iron and powdered rhenium by melting them together in the requisite proportions in crucibles of sintered corundum. The rhenium was 99·5 per cent. pure, the remainder being mostly potassium oxide which was eliminated on melting. The alloys were successfully melted in an atmosphere of hydrogen in spite of the very high melting point of pure rhenium (3440 ± 60 deg. abs.), and only when the rhenium content was more than 60 per cent. was it necessary to use crucibles of beryllia. Chemical analysis showed no marked difference between calculated and actual composition,

so there was no appreciable loss of rhenium on melting.

The alloys were studied by thermal, microscopic, and X-ray methods. Equilibrium was difficult to attain, especially with high rhenium content and in the lower ranges of temperature. Annealing treatments consisted of heating for two hours at 1300 deg. Cent. or above, eight hours at 1100 deg., and three or four weeks at 800 deg. or below. The diagram (Fig. 1) was based on thermal and other data, the temperatures given being the results of thermal analysis. From the melting point of iron

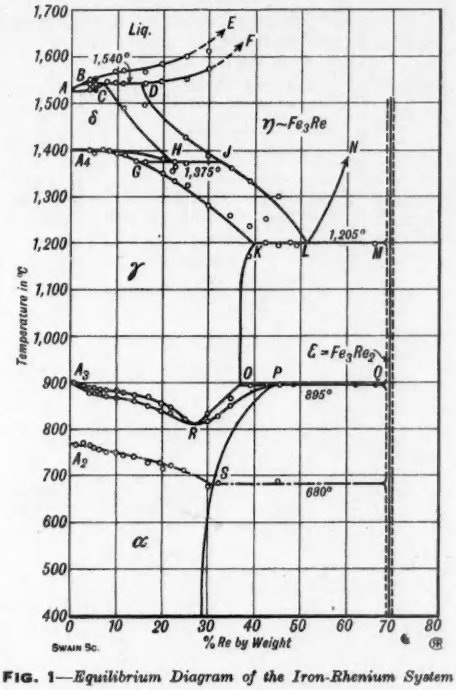

FIG. 1—*Equilibrium Diagram of the Iron-Rhenium System*

(1528 deg. Cent.) the liquidus and solidus rise with increasing rhenium content; with more than 30 per cent. of rhenium the freezing point can no longer be measured by means of a platinum platinum-rhodium thermo-couple. The phases which separate on solidification are the δ and η solid solutions, with a peritectic transformation, $\text{Liquid}_B + \eta_D \rightleftarrows \delta_C$, at 1540 deg. Cent., where B, C, and D are respectively 2·5, 7, and 15 per cent. of rhenium. The δ-phase transforms to γ at A_4, and at 1375 deg. Cent. any residual δ forms a eutectoid of γ+η. At this temperature the γ-solution holds 14 per cent. of rhenium, but the solubility increases until at 1205 deg. Cent. it reaches 40 per cent. Between 1205 deg. and 895 deg. the solubility shows little change. The transformation γ→α begins at A_3, which falls with increase of rhenium up to 27 per cent., passing through a minimum at about 820 deg. Cent., and then rises again to 895 deg. Cent. when the rhenium reaches 37 per cent. Below the line A_3R P in the diagram the α-solid solution is stable, with the A_2 magnetic change falling from 768 deg. to 680 deg. with increase of rhenium to about 30 per cent., and then remaining constant so long as any of the α-phase remains. Quenching from the γ-region was found to give a martensitic structure. The η phase is, as already indicated, deposited from the liquid. It forms a eutectoid of γ+ε at 1205 deg. Cent. and 51·5 per cent. of rhenium. For the very inadequate reason that the simplest formula of the eutectoid composition corresponds to Fe_3Re (and apparently on no other evidence) the η phase was held to be associated in some way with this compound. The origin of the ε-phase in cooling from the liquid is not known, but it was stable throughout the whole range of temperature studied, and its solubility for iron appeared to be very small. From microscopic evidence, obtained with alloys up to 66·2 per cent. of rhenium, it was estimated to contain about 70 per cent. of rhenium corresponding to the formula Fe_3Re_2. It was very hard and brittle; in fact, alloys containing a considerable proportion of this phase, *e.g.*, upwards of 45 per cent. of rhenium, were already unmachinable. At 895 deg. Cent. the alloys containing the ε-phase (*i.e.*, alloys containing 37 to 68 per cent. of rhenium) undergo a peritectoid transformation $\gamma + \varepsilon \rightleftarrows \alpha$, which is of a sluggish character, subject to a marked temperature lag in either direction. The phase boundary

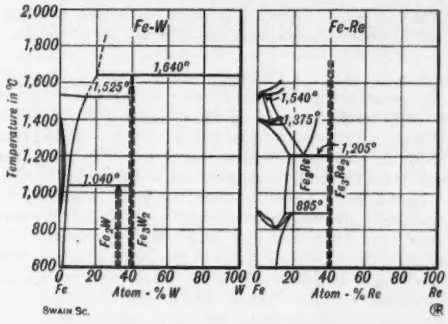

FIG. 2—*The Iron-Tungsten and Iron-Rhenium Systems*

between α and α+ε is at 45 per cent. of rhenium at the peritectoid temperature 895 deg. Cent. It falls to about 32 per cent. at the magnetic change point, 680 deg. Cent., and to under 29 per cent. at 400 deg. and below.

In the periodic classification, rhenium (atomic number 75) lies between tungsten (74) and osmium (76). Little is known of the osmium-iron alloys except that the α-phase persists up to about 11 per cent. of osmium, and that they resemble nickel-iron alloys in the pronounced hysteresis associated with the γ→α transformation.[3] The constitution of the tungsten-iron alloys is more accurately known. The tungsten-iron and rhenium-iron diagrams are shown side by side in Fig. 2. Tungsten forms a closed γ-loop, the α-phase being stable from atmospheric temperature to the melting point. The many times broader γ-region in the rhenium-iron alloys overlaps the region in which the ε-phase is stable, and the α and δ-phases are thus separated from one another by a wide temperature interval. The systems resemble one another in the formation of similar compounds Fe_2W_3 and Fe_3Re_2, both characterised by great thermal stability. There is, however, no evidence in the rhenium series of a compound corresponding to Fe_2W, which is said to exist up to

1040 deg. Cent., or perhaps to a higher temperature in a range of tungsten-iron alloys, and which corresponds to the Fe_2Ta formed by the neighbouring element tantalum.

Dr. Eggers has effectively brought together other related diagrams in the paper already mentioned,[1] and it is evident that a comparison of well-established constitutional diagrams on the basis of the periodic classification is of great importance not only in indicating features which may want confirmation, but also in checking the accuracy of generalisations about the alloying behaviour of metals in relation to their atomic structure.

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Application of Electron Diffraction to Metallurgical Problems

By A. G. QUARRELL, University of Sheffield

IN 1924, when de Broglie[1] suggested that there was associated with any moving particle a wave system which, in effect, governed the motion of the particle, it could hardly have been foreseen that he was laying the foundations upon which a new method of structural analysis would be built. Three years later, however, Davisson and Germer[2] showed that slow electrons could be diffracted by the surface of a single crystal of nickel, and almost simultaneously Thomson and Reid[3] published photographic proof of the diffraction of high-speed electrons on passing through an exceedingly thin film of celluloid. Whilst the difficulties involved in the method employing slow electrons have greatly limited its development, even the earliest experiments with electrons accelerated through potentials of more than 30,000 volts indicated the immense possibilities of the method for determining the structure of films far too thin for examination by X-rays. Until 1930 electron diffraction examination could only be applied to films less than 10^{-6} cm. thick, but the discovery[4] that diffraction patterns were yielded by polycrystalline specimens grazed by the electron beam enabled surface films only a few atoms thick to be directly examined.

Largely owing to the work of the research schools led by Thomson and Finch, the method has developed to such an extent that it can no longer be regarded as a purely academic research weapon. Indeed, quite a number of diffraction cameras have already been installed in this country for the investigation of industrial problems connected with surface structure, and there can be little doubt that, in the future, electron diffraction will find increasing use in industrial research laboratories. The intensive research carried out in our universities has led to the design of a standard diffraction camera which is readily adaptable to the investigation of almost any problem coming within the scope of the method, and which has proved so satisfactory that the design has remained fundamentally unchanged for the last five years. This, the Finch camera, is shown diagrammatically in Fig. 1, whilst a photograph of a similar camera complete with pumping equipment is given in Fig. 2. As will be seen from Fig. 1, the camera consists of a cold cathode discharge tube in which the electron beam is generated, a collimating chamber in which the beam is reduced in cross section and concentrated by means of the axial magnetic field of an adjustable focusing coil, a diffraction section in which the electron beam is allowed to impinge on a specimen suitably supported on an adjustable specimen carrier, and finally a camera section which, besides permitting visual observation of the diffraction pattern on a fluorescent screen, also enables it to be recorded directly on a photographic plate.

Although the gas pressure in the discharge tube must be of the order of 10^{-3} cm. to 10^{-3} cm., the rest of the apparatus should be highly evacuated, and this

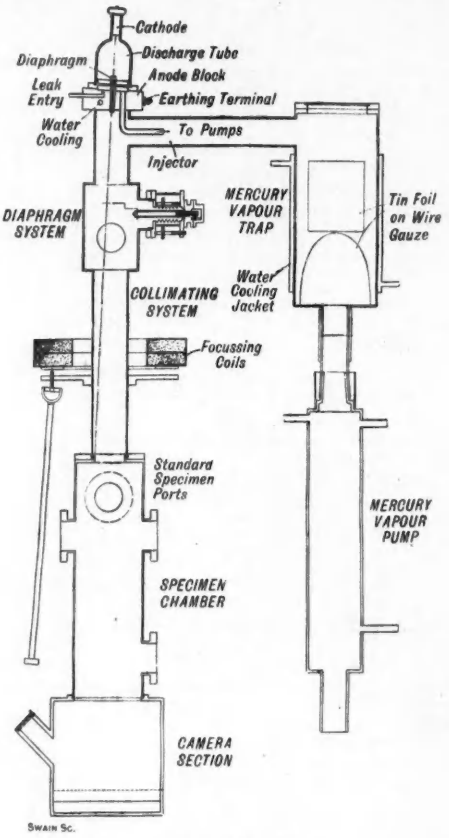

FIG. 1—*Electron Diffraction Camera*

is made possible by the use of either oil or mercury diffusion pumps, backed by a high-speed rotary oil pump. The high-vacuum technique is based upon the use of plane metal surfaces ground to a matt finish with coarse carborundum paste and lubricated with a low vapour pressure grease prepared from petroleum jelly and crêpe rubber. Constant current is fed from the high-tension supply to the cathode by means of a series diode valve run under conditions of saturation, and this, together with the constant pressure maintained in the discharge tube by means of a controlled leak system, enables a constant accelerating potential to be achieved. By adjusting the current passing through the focusing coil the electron beam can be focused on the fluorescent screen, whilst tilting the

coil not only enables the beam to be deflected into any desired direction, but also spreads it into a magnetic spectrum. Those electrons which have lost no energy by collision with the anode diaphragm or with gas molecules in the discharge tube are concentrated in the tip of the spectrum, whilst electrons of lower energy form a "tail," which is prevented from falling on the specimen by a diaphragm immediately above it. Thus only electrons of a given wavelength contribute to the diffraction pattern and any spreading of the diffraction rings observed is due to small crystal size.

In the transmission method the specimen in the form of a thin film is supported on fine-meshed wire gauze and the electron beam is passed normally through it. With a random polycrystalline specimen

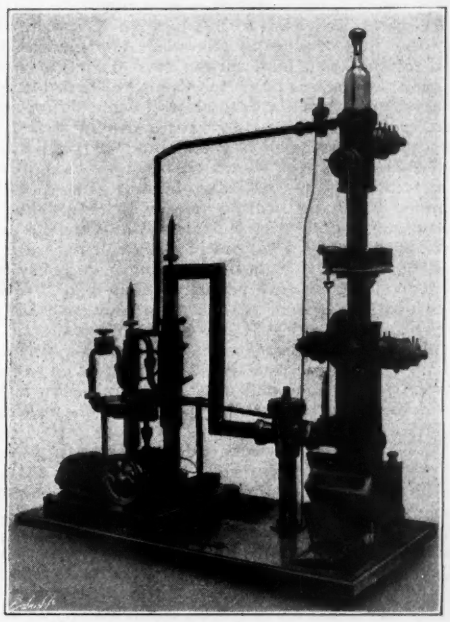

FIG. 2—*Diffraction Camera Complete with Pumping Equipment*

the pattern consists of a series of concentric rings, Fig. 3, which, however, degenerate into arcs when the crystals are tending towards a preferred orientation. The patterns obtained from the surface layers of a massive polycrystalline specimen examined at grazing incidence are actually due to transmission through the sub-microscopic needle-like crystals which project out of the apparent surface, but, because the bulk of the specimen is opaque to electrons, rather more than half of the pattern is cut off by the specimen shadow, Fig. 4. From the ring radii, camera length and the potential through which the electrons have been accelerated, the plane spacings of the crystals constituting the specimen can be determined and the corresponding structure deduced. It was first believed that the mechanism of diffraction was the same for electrons as for X-rays, but it is now known that polycrystalline specimens behave as a collection of two-dimensional lattices as far as electrons are concerned, whereas all X-ray diffractions can be accounted for on a three-dimensional theory.[4] It is partly for this reason that the exposure times in electron diffraction are only of the order of a few seconds, and the patterns can usually be seen on the fluorescent screen.

Partly because of the two-dimensional nature of the

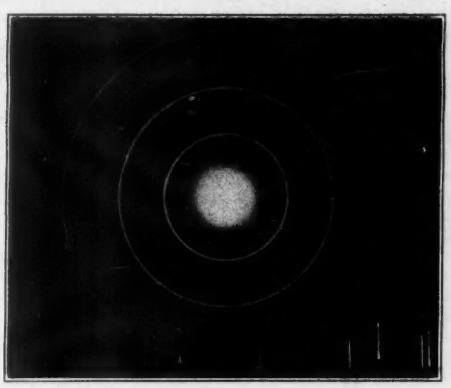

FIG. 3—*Transmission Pattern of Ammonium Chloride (Finch)*

diffraction, and partly because of the low angles of diffraction involved, the nature and extent of any preferred orientation is readily obtained from the electron diffraction pattern. This is a major advantage of the method, since thin films and surface layers frequently exhibit some degree of orientation and the nature or absence of such orientation may be the

FIG. 4—*Reflection Pattern of Fe_3O_4*

deciding factor as to the suitability of a surface for a particular purpose. It has been shown,[5] for example, that whilst random films of sputtered platinum are usually catalytically active, orientated films are invariably inert. Again, a random film of graphite may act as an abrasive and will afford little or no protection against corrosion, whilst a highly orientated layer, such as is obtained from a colloidal

suspension, is an excellent lubricant and will prevent or reduce chemical attack.

Hitherto electron diffraction has rarely been used for a systematic study of a specific problem. Instead, most of the work has been carried out with a view to increasing our knowledge of the mechanism of electron diffraction or to demonstrate the type of problem which comes within the scope of the method. Consequently, few of the examples quoted by way of illustration will have been exhaustively treated and many may even appear to have no immediate practical significance.

Thin films and surface layers are easily prepared by cathodic sputtering, electro-deposition, or by evaporation *in vacuo*, and these methods have been frequently employed in the preparation of specimens for electron diffraction. A number of interesting observations have been made in consequence. Thus, indirect methods of investigation, such as measurements of electrical resistance, suggest that sputtered or evaporated metal films are amorphous, but the general experience with electron diffraction is that all such films are crystalline. In view of this, it must be concluded that the high resistance values observed with such thin films are to be attributed to the existence of more or less isolated areas of crystalline material rather than a continuous film of amorphous metal. Again, a film sputtered from a cathode of gold and platinum wires twisted together consists not of a gold-platinum alloy, as might be expected, but of a mixture of the two metals. This suggests that atoms prefer to attach themselves to nuclei of the same metal, and, furthermore, emphasises the considerable surface mobility possessed by the atoms in very thin films. Similar experiments gave an early indication of the effects which the substrate might exert upon a deposited film. Thus, Thomson[6] showed that silver electro-deposited on an etched cube face of a copper single crystal, followed the orientation of the copper. Similarly, Finch, Quarrell, and Wilman[7] have sputtered gold, under conditions favourable to the production of a random film, on to a glass disc, one half of which was already covered with sputtered platinum in (111) orientation, and found that the gold over the platinum had taken up a similar orientation, but was random on the glass.

In addition to the orientation effect, the substrate may exert a considerable influence upon the lattice dimensions of the deposited film, or even determine its crystalline structure. For example, although aluminium is normally a face-centred cubic metal, it has been shown by electron diffraction[8] to form tetragonal crystals when vaporised on to a crystalline platinum substrate. Owing to the fact that the platinum was in (100) orientation, *i.e.*, a cube face of each crystal was parallel, or nearly so, to the specimen surface, and since this orientation was imposed on the aluminium, it was possible to show that, in the plane of the specimen, the aluminium had adopted the lattice dimensions of platinum, whilst in the direction normal to this it had retained its characteristic distance of atomic separation. The effect was only detected in the thinnest films, presumably because the substrate influence diminished with increasing thickness. In the same work it was shown that the initial oxide film formed on a clean zinc surface prepared *in vacuo* was also basally pseudo-morphic with the underlying metal crystals. Shortly afterwards[9] the effect was confirmed, and it was further shown that the pseudo-morphic oxide was a continuation of the zinc lattice which prevented further exposure of the zinc to the atmosphere. Breakdown of the pseudo-morphic oxide, however, produced very much smaller random crystals of the normal oxide which offered little resistance to the passage of oxygen, so that a new layer of the protective oxide was formed. These results enabled Vernon[10] to account for previously unexplained features of the weight-increment/time curves which he obtained in a study of the oxidation of zinc by a gravimetric method.

In general, any surface which has undergone mild chemical attack is suitable for use as a reflection specimen, and many examples of surface films formed in this way and subsequently identified by electron diffraction can be quoted. Thus it has been shown that the nickel selenide film formed when selenium vapour is passed over heated nickel does not crystallise in the hexagonal form of the normal NiSe, but in a pyrites structure which has been attributed to $NiSe_2$. Again, when copper is incorrectly degreased with trichlorethylene it becomes coated with an invisible film of cuprous chloride. The blue oxide film formed on a high carbon steel during a commercial process was much too thin to be detected by X-rays, but yielded a very clear electron diffraction pattern of FeO. In a recent paper dealing with the atmospheric oxidation of metals and alloys, Bound and Richards[12] have shown that brasses containing less than 30 per cent. zinc become coated with cuprous oxide on heating in air at temperatures below 300 deg. Cent., whilst one containing 38 per cent. of zinc gives a pattern of zinc oxide on heating at 250 deg. Cent. Thin films of copper on collodion were found to be only partially oxidised even after nine months' exposure to air at room temperature, but one hour at 100 deg. Cent. completely oxidised a similar film to cuprous oxide. Preston and Bircumshaw[13] have shown that the air-formed film on aluminium is amorphous and does not recrystallise until it has been heated to temperatures near the melting-point of aluminium.

The application of electron diffraction to the study of electro-deposits has made it clear that the nature of the substrate is of the utmost importance in determining the adhesion of the plating. It might well be expected that alloying would take place between the cathode and the deposited metal, but no signs of alloying during deposition have been detected, even in the first few layers of atoms, although chemical displacement from solution of one metal by another invariably results in alloying. Finch and Sun[14] showed, however, that whilst with a polished substrate the crystal size and orientation of the deposit was determined by the bath composition, temperature, and current density, if, on the other hand, the cathode possessed a crystalline surface structure, it could exert a considerable influence upon the deposit. For example, under the same conditions of deposition, chromium electro-deposited on copper formed smaller crystals than on nickel, iron, tin, or bismuth, and in a similar manner the orientation taken up by the crystals in thin layers of a deposited metal was dependent upon the substrate. It was shown that when the deposit crystals are able to follow the crystal size and orientation of the substrate, good adhesion usually results. In certain special cases the deposit crystals may not only share a common plane of orientation with the substrate, but, in addition, a given crystallographic direction in the deposit crystals may be parallel to a given atom row in the basis metal. Thus, in the case of iron on gold, the iron crystals not only share the (100) or cube face orientation of the gold, but also arrange themselves so that cube edges

of the iron crystals are parallel to the cube face diagonals of the substrate crystals. The reason for this is that the side of unit cube of iron is $1/\sqrt{2}$ times that of gold with the result that the positions adopted by the first layer of iron atoms deposited can be exactly the same as those which would be assumed by gold atoms, and yet at the same time these iron atoms are in their correct positions in the cube face plane of the iron lattice. In other words, the first layer of iron atoms can be regarded as common to both gold and iron lattices. It is hardly necessary to add that such iron films showed excellent adhesion, because they were in effect atomically welded to the gold base.

An interesting phenomenon was observed[15] when a random nickel surface was made one electrode in a $2\,N\text{-}H_2SO_4$ solution and an alternating potential of 12 volts was applied between it and a carbon electrode. After this treatment the surface crystals of the nickel possessed almost perfect (110) orientation, and were clearly in the most suitable condition for exerting a strong orienting influence on any subsequent deposit.

It is the general experience that rolled or beaten foils of face-centred cubic metals invariably exhibit cube-face orientation, and Burgers and Ploos van Amstel[16] have shown that with rolled tungsten the cube faces of the body-centred cubic metal are in the plane of the film, and, in addition, the (110) planes are parallel to the direction of rolling.

Other problems to which the electron diffraction method has been applied are amalgams, semi-permeable membranes, oxides on molten metals, indirectly heated cathodes of wireless valves, the Beilby layer, wear and "running-in," lubrication, and so on. In addition to such applications, a great deal of work has been done with single crystals as specimens. The resulting patterns are exceedingly complicated, and their analysis is by no means simple, but such work can be truly classified as academic research, since it has as its object not the solution of specific industrial problems but the elucidation of certain features of the mechanism of electron diffraction which are at present imperfectly understood.

In conclusion, it may be said that the development of electron diffraction has reached the stage at which there is no good reason why it should not be applied more generally to the investigation of surface structure problems. In metallurgy, its potentialities in the investigation of corrosion and the structure of metal coatings are already obvious, and the systematic application of electron diffraction in such fields must necessarily lead to a fuller understanding of the phenomena involved.

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Magnesium Alloys: Application and Future Development

No. II (*continued from February issue*)

MAGNESIUM ALLOYS IN MACHINE CONSTRUCTION

A VERY comprehensive and informative account of the application of magnesium alloys in machine construction is given by W. Walbersdorf, Berlin.[2] The general aim is to use as little material as possible for a mechanical component, yet to use sufficient to withstand the highest forces present. The use of magnesium renders this possible in many ways, and the result is an advance in technique. The account referred to describes the position largely from the German standpoint, depending, as it does, on the internal economic situation. Magnesium alloys often take the place of other metals, especially aluminium and sometimes iron.

A classification is given below in which die and pressure die castings are not included because it is necessary to know how many are made in order to assess their economic value, and such large quantities are not used in machine construction.

The following examples show the possibilities in the application of magnesium alloys.* It is advisable in the present stage of development only to use the alloys for the less highly stressed components :—

Steam Engines (Stationary).—Excentric ring, sand casting alloy A 9 V with heat treatment; spring plate for distributing valve motion, pressed and wrought alloy AZ 855; oil sumps, sand casting alloy AZ 31 or sand casting alloy CMSi.

Ships' Engines.—Swing lever for operating auxiliary machines, pressed and wrought alloy AZ 855.

Transport.—Crank cases for steam engines, sand casting alloy AZ 31; cover for crank case, sand casting alloy AZ 31 or CMSi.

Compressors.—Crankshaft cover for two-stage compressor, closing plate and cover for slide-way where these must often be opened for inspection, sand casting alloy AZ 31 or CMSi; cover to crank case and crank case for movable compressor, sand casting alloy AZG.

Refrigerating Machines.—Cover and crank case for small refrigerating machines, sand casting alloy A 8 K; toothed wheel pump housing, sand casting alloy AZG.

Chemical Apparatus.—Bearing for stirring gear, serving rod bearings for sand casting alloy AZG.

Steam Turbines.—Regulating rods, pressed and wrought alloy AZ 855.

Lathes, Drilling, Planing, Milling Machines, &c.—Gear-box covers, cover for spindle box on lathes where these are to be frequently opened and where it is desirable to reduce noise, sand casting alloy AZG; gauge bodies for setting-up appliances.

As a result of the good thermal properties (conduction) of magnesium alloys, it is possible to make oil sumps of this material for high-speed engines. The usual oil cooler may be omitted in certain cases since the cooling action of the air on the sump suffices to conduct away the heat of the oil. In the case of stationary engines, efficient cooling air may be obtained as a result of the motion of the fly-wheel in which case it is necessary to provide sheet channels, &c., to guide the air from fly-wheel to sump.

* The properties of the alloys referred to were described in the first part of this paper which appeared in the February issue.

Magnesium sand casting alloy AZF or pressed or wrought alloy AZ 855 may be used for crosshead bodies provided the stresses are not too high. The advantage of a reduction in mass is obvious in many ways in the case of rapidly acting machines. For low specific loads, as for crossheads for excentric rod guides in valve gear for steam engines, it is possible to make the whole of the crosshead of magnesium alloy with good bearing properties, or even to provide the crosshead body with a shoe slide of an alloy of better rubbing properties. Further, it is possible to make connecting-rods and excentric rods of magnesium alloy for small specific loads.

Since inertia is thereby reduced, especially with quickly running machines, there is a corresponding reduction in the load on the bearings and in the forces on the connecting-rods. With water-cooled cylinders in magnesium alloy, the possibility of corrosion can be avoided if a cartridge containing a substance protective against corrosion, such as alkali-bichromate, be introduced into the cooling water system. The cartridge life is given as three-quarters of a year. The cooling media in general use to-day have a corrosive effect on these alloys, and this excludes their use for components which come into contact with the coolant in refrigerators.

As with most metals, there are certain features to be taken into account by the designer wishing to incorporate magnesium alloys in his constructions, and these will be briefly referred to now.

In the case of sand castings the wall thickness should not be less than 4 mm. Since magnesium alloys are sensitive to conditions of stress concentration, the change from one section to another section of different size must occur gradually. Since the modulus of elasticity amounts to only 4000 to 4500 kilos. per square millimetre, certain factors must be borne in mind. Thus the resonance range of constructional components lies below that for aluminium alloys and heavy metals of similar design. Further, magnesium alloys are apt to distort rather easily under load as a result of the low modulus. It is therefore advisable to support the surrounding metal at the edge of bolt holes and other places subject to stress concentration by a thickening up of the section. In general, it is a good plan to aim for a box-like form of section which possesses a large moment of resistance.

As a result of the elastic behaviour of magnesium alloys, constructional parts are less sensitive to sudden blows or impacts and they possess a greater capacity to absorb work. Should serious stress concentration occur by screwing up on assemblage, the low modulus of elasticity allows the component to accommodate itself, and the resulting stresses are less than they would be for other metals. The power also to absorb noise is useful for gear housings and covers. Where ball bearings are to be inserted, it is best to introduce a steel bush to accommodate the bearing. For bolts often to be unscrewed it is better to put heavy-metal thread inserts in the castings. This may be done in several ways.

The resistance of magnesium alloys to rubbing action is low. In spite of this, cam bearings have, it is stated, proved satisfactory when made of this material for English and American aero-engines. Oil pump housings for toothed wheels are generally so constructed that the wheel shafts run in the housing itself without the necessity of intermediary bushes. Naturally, the better the surface of the shaft the better the operation of the bearing. Given an adequate supply of oil, the permissible face pressure is 1·5. to 2 kilos. per square centimetre for cast alloys, and 3 to 4 kilos. per square centimetre for wrought alloys provided the temperature does not exceed 100 deg. Cent. Bushes must be inserted if these conditions cannot be fulfilled.

In the case of Elektron castings, operating temperatures should not exceed 200 deg. Cent. They should not exceed 250 deg. Cent. for the wrought alloy AZ 855. The strength values decrease about 10–30 per cent. up to these temperatures, depending on the alloy, whilst at below 0 deg. Cent. the values increase somewhat.

Components subjected to centrifugal forces should be so manufactured that the forces act in the general direction of the fibre of the metal. According to the direction of the force to the direction of the fibre, the strength of forgings may vary up to as much as 50 per cent. Since castings have a dense structure internally they are suitable for housings of all kinds. For components subject to elevated temperatures, it may be necessary to take thermal expansion into account. This depends very much on the alloy used. The expansion is about 10 per cent. greater than for aluminium casting alloys and about twice as much as irón.

Shrinkage on casting depends on the form of the casting and varies from 1 to 1·4 per cent. It is 1·9 per cent. in the case of CMSi and AM 503. Casting temperatures vary from 700 deg. to 800 deg. Cent. and forging temperatures from 300 deg. to 380 deg. Cent., the highest permissible temperature being 420 deg. Cent.

Magnesium alloys are sensitive to the corrosion attack of many substances. Where they come into contact with water they should be given a layer of varnish. Where a varnish is not likely to stand up to vibration in service or to the impingement of foreign bodies such as sand which rub it away, then magnesium alloys should not be used and Hydronalium (an aluminium-base magnesium alloy) is recommended as a substitute. The use of a cartridge containing protective substances for insertion into cooling water has already been mentioned, and alkali-bichromate pickle produces a very effective protection on the alloys. Intercrystalline corrosion has not been observed. Magnesium alloys are attacked by glycol, glycerine, acid and salt solutions, while alkaline solutions have no action at all below 120 deg. Cent. It may be necessary to isolate a component in magnesium alloy from its neighbour by a layer of varnish, a sheet of oil paper, or bakelite, though such a procedure is not always necessary and even an oil film is often quite sufficient to protect the metal from corrosion. It is noteworthy that Elektron may be exposed to atmospheric conditions for many years without corrosion taking place; actually, a thin protective surface layer is formed, which is sufficiently protective to prevent further attack. Under similar conditions of exposure iron would be corroded to a much greater extent.

In riveting it is usual to use rivets of copper-free aluminium alloy. If rivets of other metal are used, then insulating materials are used. This applies specially where riveted jobs are exposed to atmospheric moisture. Where the two parts to be riveted come into contact, they should be painted with a layer of varnish or sheet of insulating material as a protection against corrosion.

Magnesium alloys are readily welded. The alloy AM 503, especially if in the form of sheet, is particularly suited for gas welding. The length of the welded part may be made as long as desired, but in the case

of AZM sheet it should not exceed 200 mm. Special fluxes are necessary during welding, and they must be carefully removed from the joint afterwards. The wire used for welding sheet should be of the same composition as the sheet, and the best welds are obtained only when the two parts to be joined consist of the same alloy. Ribs or bosses may be welded quite easily to castings; care should be taken to heat up the casting to 270 deg. to 330 deg. Cent. slowly in a furnace before attempting to weld, and this temperture should be maintained during the operation. Mechanical tests on the welds show a strength equivalent to that of the material joined. The strength of the welded part may be increased by hammering the actual joint which results in a refinement of the grain of the metal. Also, any defective localities in a casting can be repaired by welding. Resistance welding has been successfully applied where parts have to be welded in series. Pressure welding can be used under certain conditions. In this case, thin zinc foil a couple or so hundredths of a millimetre thick is placed between the parts to be united, and the whole heated to 350 deg. Cent., whilst the pressure applied should not be less than 500 kilos. per square centimetre.

Cold-pressing operations permit the manufacture of all kinds of parts from Elektron sheet—for example, drivers' seats and box covers. Rubber may be interlaid between the sheet and die with good results in ensuring a satisfactory pressing. The operation must be carried out at a temperature of 280 deg. to 320 deg. Cent. Bending radii should not be less than twice the sheet thickness. A new sheet alloy AM 537 has recently become available which has still better cold bending properties.

The machining properties of the magnesium alloys are excellent; in fact, better than for any other metal. Cutting speeds of 1500 m. per minute are possible in milling operations with a feed of 1·5 m. per minute. In view of the high speeds possible, special types of machine are desirable. All cutting tools must be kept sharp. Special forms are prescribed for them. It is better not to use any cooling medium at all during machining. Liquid coolant such as oil, &c., may be used on exceptional occasions. It is necessary to remove and isolate all accumulations of cuttings, particularly fine swarf, away from the machines from time to time, to avoid risk of fire. It is most important to keep water away from swarf, even in the event of a fire! Should a swarf fire arise from the use of a blunt cutting tool, then it can be extinguished by throwing upon it a quantity of cast iron cuttings. Particular attention should be paid to the adequate removal of magnesium dust produced in grinding operations which is best effected by sucking or drawing the dust into a special collecting chamber. Water-tight iron drums are very satisfactory for isolating and storing swarf and dust. There is no danger provided machinists and users are acquainted with proper safety measures. In general, there is absolutely no fire danger with Elektron. This is shown, for example, in the use of Elektron pistons for racing car engines, which are in contact with the gases of combustion.

In conclusion, the disadvantages of magnesium alloys may be summarised thus. Their strength is not equal to that of high-duty aluminium alloys; they tend to deform more owing to their low modulus of elasticity; only small specific surface pressures are permissible; resistance to wear is low compared with the heavy metals; sensitivity to stress concentrations, and lower resistance to corrosion when in juxtaposition with the heavy metals. The high price per pound is also a disadvantage, but price reduction should be possible. In this connection, considerations of strength and low specific gravity are all-important in price comparisons. A special warning is given when trying out castings in magnesium alloy formerly made in cast iron, that a fresh pattern based on magnesium casting technique must be used and the old pattern as used for the cast iron must not be employed.

The advantages of magnesium alloys, in brief, are good castability and hot working possibilities permitting the production of castings of all shapes, forgings, extrusions, &c.; tolerances for casting are small; low specific gravity is also an advantage for parts frequently handled and for the unsprung parts of vehicles, thus wear in general is correspondingly less; inertia is reduced in moving components, permitting a lighter construction; their capacity to eliminate noise in gear wheel housings; the endurance strength under bending of the magnesium alloys is high relatively and they are relatively non-sensitive to blows and impact stresses; the machining properties are excellent in every way. Such properties affect price considerations in favour of the alloys.

Recent Developments of Spectrographic Analysis

No. I

By E. VAN SOMEREN, B.Sc.

AT the annual general meeting of the Institute of Metals in March, an afternoon was devoted to a discussion on Spectrographic Analysis which was opened with a paper by Mr. F. Twyman, F.R.S., on the non-ferrous applications of this method in industry.[1]

A keen discussion followed, in which thirteen members took part, of whom five were concerned with the application of spectrochemical analysis to aluminium alloys. M. G. Gauthier said his firm (Produits Chim. et Met Alais, Froges Camarque) relied on spectrochemical analysis for the control of certain constituents of aluminium alloys such as duralumin after carrying out a statistical test of the quantitative accuracy of the method for each element. For copper and silicon, chemical methods were still used, also for magnesium above 5 per cent. where the maximum error of ±3 per cent. was excessive. He referred to many interesting practical details of procedure, such as the method of casting samples and their shape, the necessity for preliminary sparking before the exposure, and the contamination of aluminium samples by iron tools which was revealed by differences between chemical and spectrographic tests. Dr. Whalley, of the Government Chemist's Laboratory, also found spectrochemical methods sufficiently accurate for most magnesium and aluminium alloys, and quoted evidence from a thousand duplicate spectrochemical tests on manganese in aluminium to show that the mean deviation of the results was about ±5 per cent. A non-recording microphotometer and spark were used, and chemical methods were preferred for most constituents above 5 per cent., also for copper and aluminium. Dr. Frommer, of

High Duty Alloys, Ltd., also gave details of quantitative investigations of the accuracy of spectrochemical analysis, particularly for magnesium in aluminium. From a batch of 180 specimens which were analysed both spectrographically and chemically he found an average deviation between the two results of 4·5 per cent., and only one deviation of as much as 10·5 per cent. He went on to discuss the sources of error, and confirmed the statement made by Mr. Twyman that a simple resonant spark circuit is best for aluminium and that heterogeneity of samples is a very important source of error. In this connection he presented some photomicrographs and very striking data dealing with heat treatment of an alloy which led to an alteration of relative line intensities corresponding to 18 per cent. of the amount present. A further source of error in his experience was the variability of the A.C. supply voltage which was used for the primary of the spark circuit transformer. By using a controlled voltage a marked improvement in the scatter of the results from a batch of 134 samples was obtained. The necessity for preliminary sparking to stabilise conditions which was admitted by Mr. Twyman and deplored by Monsieur Gauthier was found by Dr. Frommer to have been over-estimated by other workers. He proved by statistical tests that a very brief preliminary sparking time was sufficient for aluminium alloys, and that consistent duplicate spectra could be obtained even although the relative intensity of the lines was altering during the time of exposure.[2] The effect of one minor constituent on the apparent concentration of others was also illustrated by quantitative data.

Mr. Handforth, of Birmetals, Ltd., also spoke of the industrial use of quantitative spectrochemical methods for aluminium alloys, and referred to the problem of magnesium in aluminium which could occur as Mg_2Si, as Al_3Mg_2, or in solid solution. Would the relative intensity of the Mg radiation in the spark be the same in each case ? No reply was forthcoming, but the writer is of the opinion that the characteristics of spark circuit and electrode shape may be chosen so that uniformity would be obtained, although the conditions might not be the best for obtaining sensitivity to minor impurities.

Dr. A. C. G. Gwyer, of British Aluminium Company, also spoke of aluminium alloys, and of the difficulty the industrial producer of metal had in meeting chemical specifications by spectrochemical analysis. He stated that, to deal with the spectrochemical analysis of aluminium alloys, the Bureau International d'Aluminium has recently founded a sub-committee which met in Germany recently for the first time. Interchange of information and experience is needed before such factors as type of apparatus, type of spark circuit, and size of electrode can be standardised.

Dr. H. W. Brownsdon, of I.C.I. Metals, Ltd., spoke of the value of spectrographic methods in quality control of production, and urged that there should be closer co-operation between producers and consumers of metals in agreeing on spectrographic methods of specifying the purity of materials. He suggested either that a lead should be given by Government departments who were large buyers of metal to specification, or that the Council of the Institute of Metals should set up a committee for the purpose of obtaining official recognition for this method of analysis. Such a committee might begin with the industrial alloys of aluminium, dealing later with other metals and alloys ; it would detail a suitable technique and obtain agreed acceptance of the method of test by buyer and seller. Dr. Brownsdon also expressed a wish that the name of the method of analysis should be officially standardised, and expressed a preference for spectrographical analysis rather than spectrochemical analysis, or any other alternative. Later, Mr. Handforth mentioned that the A.I.D. had offered to accept spectrochemical evidence of analysis when he could produce tests on a hundred samples by both chemical and spectrographic methods showing similar accuracy of the tests.

Professor Breckpot, of Louvain, briefly described the large-scale application of routine spectrochemical analysis in two factories. In one it was used for controlling the purity of metals, and only visual methods of line-intensity comparison were found necessary, since the experienced eye is an excellent photometric instrument. Estimations of bismuth in lead made in this way sufficed to show up an error in the chemical method used for the estimation of bismuth. In the analysis of pure tin the saving of material which resulted from using spectrochemical methods instead of chemical tests on large samples amounted to several hundredweight per month.

In another factory a spectrograph was installed in a laboratory independent of the chemical one, and worked for some months on samples of the same materials, the two sets of results being compared only by the central office. This led to the detection of several systematic errors in the analytical methods, principally on the chemical side. A stepped logarithmic sector and microphotometer was used for accurate measurements on arc spectra, and gave results of satisfactory accuracy by a quick method. By the use of a longer method in which complete blackening curves were plotted for each pair of lines being compared, results could be obtained of the highest accuracy (± 2 per cent.) which were practically independent of such photographic variables as time or temperature of development.

Dr. Judd Lewis, who, in his practice as a consultant, has used spectrographic analysis for over twenty-five years, spoke of its value to the occasional user as well as the routine worker and said he had found it sufficiently accurate to solve problems of disagreement between two analysts where he had been called in as referee. By using synthetic powder samples in the arc, visual examination of the spectra could be made to give remarkably accurate results, which could be further improved by the aid of photometry.

Mr. M. Milbourn, of I.C.I. Metals, Ltd., also contritributed as a user of arc methods for quantitative analysis, and told of the means used to overcome some of the sources of inaccuracy attributed to the arc by critics. He said that selective distillation of impurities during exposure can be avoided for nearly all copper alloys by choosing suitable standard conditions, but was a possible source of error in the determination of silicon and phosphorus. The background of the arc spectrum appears to be largely due to the atmosphere, and is radiated chiefly by the central portion of the arc. It is reduced considerably by using a very short arc, and the lower limit for the detection of arsenic in copper can be extended from 0·02 to 0·001 per cent. by using an arc only 1 mm. long, or by otherwise increasing the density of copper vapour in the arc, as might be done by increasing the current.

Variations in size of sample affect the arc as well as the spark by causing variations in the relative

intensity of impurity lines compared with those of the main constituent, and it was found in practice that there seemed to be a parallellism between this relative intensity and the brightness of the arc. If differently shaped samples of the same material are used as electrodes, and the arc current is adjusted to give the same brightness as measured by a photo-electric cell, uniform spectra were obtained.

Dr. Barr, of the N.P.L., had experience of the use of spark, arc, and flame methods in spectro-chemical work, more for qualitative than for quantitative analysis. He referred to one or two difficulties in the use of the first two methods, and gave more details of the flame method, in which the material to be analysed is dissolved and the solution is introduced by spraying into the oxygen supply of an oxy-acetylene flame. For the limited number of elements to which this method is sensitive, the very clean spectra obtained facilitate quantitative work. Dr. Barr described an all-glass spray apparatus, which could be made in batches, all of which would give the same rate of feed of solution into the flame.

Mr. Twyman, in closing the discussion, said that it had served its purpose in leading to interchange of technical information between users of spectro-chemical methods, and that he had himself been pleased to learn something new from every contributor.

During the discussion several speakers referred to a recent compilation called "Spectrographic Analysis in Great Britain," edited by A. C. Candler.[3] This small book consists of reports from twenty-eight users of spectrographs on the range of materials to which they apply spectro-chemical analysis, and in some cases also on the methods which they employ.

Leading contributions to this collection come from the various Government Services, and that from the Admiralty Inspection Laboratory has the greatest engineering interest. Great reliance is placed on spectro-chemical methods in this laboratory, and they are applied to the estimation of lead, bismuth, antimony, arsenic, iron, nickel, silver, aluminium, manganese, tin, zinc, and cadmium in non-ferrous alloys. The method is also applied to the routine testing of a wide range of added elements in both plain carbon steels and the more complex alloy steels, for example, in the nickel-chrome, molybdenum, and vanadium steels, silicon, manganese, titanium-cobalt copper, and aluminium are estimated spectrographically, besides the four elements already mentioned. The technique adopted at Sheffield is very rigidly standardised, and for steels triplicate exposures are made in order to average out the deviations due to heterogeneity of the material.

The tests reported by the War Department Chemist apply to a wide range of ferrous and non-ferrous metals, and as the technique for these is well known, more space is devoted to a discussion of spectrographic tests made on paint and other non-metallic materials whose quality must be closely controlled. In testing paint, it is incinerated, the ash mixed with ferric sulphate and ammonium sulphate, and compressed to a pellet which is then volatilised in an arc between copper electrodes. Comparison of the relative intensity with certain iron lines is then made the basis of a quantitative analysis.

Four large cable manufacturing companies report on the utility of spectro-chemical tests for checking the purity of lead and its alloys, as well as of other substances, both metallic and organic, which are used in cable manufacture. The Post Office Research Station, which was a pioneer organisation in the recognition of spectro-chemical methods for lead alloys, now also applies the method quantitatively to zinc, tin, and antimony, as well as qualitatively to a wide range of metals. The identification of metals or alloys used in small electrical contacts is typical of their work.

The Government Chemist's Laboratory is among those which use the spectrograph for aluminium alloys, as mentioned above.

About a quarter of the book deals with the applications of spectro-chemical methods to materials, such as glasses, paint, fabrics, food, soil, and plants, which are not of direct metallurgical interest, but some of this work is of considerable intrinsic interest, and advances in spectrographic technique which were first developed for mineralogical purposes frequently prove to be also applicable to metallurgical analysis. One example is the method in which a solution is sprayed into a flame. This was originally tried in order to overcome the difficulty of analysing non-conducting materials, but is now also used for alloys where heterogeneity or the disturbing effect of one added element on the estimation of another make ordinary spark testing insufficiently accurate.

The book closes with a contribution from a forensic science laboratory, discussing the special problems which the spectroscopist has to deal with in analysing clues such as dust, paint marks, or filings.

The general impression given is that chemists are working in close co-operation with spectroscopists to take advantage of the saving in time and material which can result from spectro-chemical methods. One user of a spectrograph records a preference for colorimetric chemical methods for the quantitative estimation of traces of ingredients, but the remaining comparisons made in the book between chemical and spectrographic methods are mostly in favour of the latter.

REFERENCES

[1] This paper was reprinted in THE ENGINEER, March 24th, page 387.

[2] This confirms the writer's own experience in 1933, *J. Inst. Met.*, LV 267, 1934.

[3] Published by A. Hilger, Ltd., 1939.

Books and Publications

Metals. By Sir HAROLD CARPENTER and J. M. ROBERTSON. Royal 8vo, 2 vols., pages xi+xii +1485. 1939. London: Oxford University Press. £5 5s.

IT may confidently be expected that this book will long occupy an important place among metallurgical publications, because it describes the whole field of physical metallurgy in the form of a coherent system as it appears to the authors. It is essential to the success of such a plan that it shall be conceived on a broad scale and that its authors can be trusted to present an accurate and well-balanced survey giving full weight to every aspect of the subject—two requirements, the fulfilment of which in this case no one will dispute.

The general object of the book is to describe the characteristics of metals, and to explain how they are related to composition and treatment on the one hand, and to properties on the other. It is

divided into six parts, each of about 200 pages, except Part V (Industrial Ferrous Alloys), which is about twice as long as the others. Part I describes the characteristics of pure metals, their structure and behaviour under stress at different temperatures. Part II is devoted to the theory of alloys; Part III to mechanical properties and resistance to oxidation and corrosion. Part IV is concerned with "treatment" in the widest sense of the word, including casting, solidification, and consequent segregation, heat treatment (including the effect and control of furnace atmospheres) and mechanical treatment, such as rolling, forging, extrusion, drawing, pressing, &c. These parts constitute the first volume. The second volume consists of Part V, dealing with the ferrous alloys, and Part VI with the non-ferrous metals and alloys of industrial importance. It should be added that these subjects are treated in the light of the latest developments of metallurgical research and industrial practice. The whole book is written in such a way as to make it possible for anyone with some scientific or technical training, but without any previous knowledge about metals, to begin at the beginning and build up an ordered and comprehensive knowledge of the subject as he reads. It is regrettable that the high price will prevent the average student making it a constant companion of his studies. It would, in a sense, be true to say that this is not a book for the metallurgical specialist. He will not here find the documented mass of information which is the essential basis upon which much of his work must be built. Yet in another, and wider, sense it may be regarded as the one existing book on metallurgy that every specialist, however narrow his field, should read and become thoroughly acquainted with. It emphasises the essential unity of the science of metallurgy whatever direction its applications may take, and in some very lucid and readable sections directs attention to the bonds which link metallurgy more closely than ever with modern developments of physics and chemistry.

The authors are to be congratulated on having completed a work which may influence the trend of metallurgical education and progress for many years to come.

Alloys of Iron and Nickel: Vol. I, Special-purpose Alloys. By J. S. MARSH. 8vo, pages xii+593. 1938. New York and London: McGraw-Hill Publishing Company, Ltd. 36s.

THIS is the first of two volumes, in the well-known "Alloys of Iron Research" Monograph Series, dealing with the alloys of iron and nickel. It is devoted to the constitution and properties of high-purity iron-nickel alloys and particularly to the properties of alloys used for special thermal, electrical, and magnetic purposes. Of these there is an enormous variety. Nickel is a component of the alloys which have the highest and the lowest values of magnetic permeability. One nickel alloy has the highest known coefficient of thermal expansion among ferrous materials, another the lowest value for any material. The amazing range of properties covered by the nickel-iron alloys ensures for them a position of foremost importance, both industrially and scientifically. In this book the physical properties of the iron-nickel and related alloys are described and discussed in the light of their constitution and metallurgical treatment, but with sufficient comprehensiveness to be useful to the physicist and electrical engineer. It is mainly on the electrical side of engineering that the materials under consideration in this volume are applied—to the telephone, telegraph, and radio services, and to power transmission. The final chapters deal with mechanical properties and corrosion resistance of the alloys. The properties of structural materials, nickel steels, and cast irons, will be dealt with in Vol. II; but even to those who are mainly interested in this side of the subject Vol. I is also indispensable as it contains a full account of the equilibrium diagrams of the iron-nickel, iron-carbon-nickel, and more complex systems which are essential to the interpretation of the constitution of the nickel alloy steels.

The book is planned on the lines already made familiar, and in wealth of information and critical assessment of its value maintains the high standard set by previous monographs of the Alloys of Iron Research Series.

The Principles of Metallographic Laboratory Practice. By G. L. KEHL. 8vo, pages xi+359. 1939. New York and London: McGraw-Hill Publishing Company, Ltd. 21s.

THIS book has been written with the object of bridging the gap between theoretical instruction in physical metallurgy and practical laboratory technique. Without presenting any definite course of laboratory exercises, it contains a statement of underlying principles, together with explicit recommendations of procedure to be adopted, and equally important warnings of procedure to be avoided.

The field covered includes micro and macro-examination, photomicrography (with the addition of a special chapter on the principles of photography), hardness testing (but no other mechanical tests except the cupping test), pyrometry, thermal analysis, and a variety of "special metallurgical tests," such as grain-size measurement in steels and non-ferrous materials, the McQuaid-Ehn test, and the Shepherd penetration-fracture test for hardenability. Reference tables included in the appendix, especially those dealing with etching reagents, are a valuable feature of the book. The treatment of all questions related to structure appears to be more thorough and satisfactory than that accorded to other methods of investigating constitution, *e.g.*, thermal analysis. Though essentially a book to aid students of metallography, it contains much information which should be helpful to those who are called upon to employ microscopical and allied methods of examination in the solution of their practical problems.